



The  
Patent  
Office

PCT/GB 99/01467

GD 99/1467

INVESTOR IN PEOPLE  
The Patent Office  
Concept House  
Cardiff Road  
Newport  
South Wales  
NP10 8QQ

REC'D 01 JUL 1999

WIPO PCT

I, the undersigned, being an officer duly authorised in accordance with Section 74(1) and (4) of the Deregulation & Contracting Out Act 1994, to sign and issue certificates on behalf of the Comptroller-General, hereby certify that annexed hereto is a true copy of the documents as originally filed in connection with the patent application identified therein.

In accordance with the Patents (Companies Re-registration) Rules 1982, if a company named in this certificate and any accompanying documents has re-registered under the Companies Act 1980 with the same name as that with which it was registered immediately before re-registration save for the substitution as, or inclusion as, the last part of the name of the words "public limited company" or their equivalents in Welsh, references to the name of the company in this certificate and any accompanying documents shall be treated as references to the name with which it is so re-registered:

In accordance with the rules, the words "public limited company" may be replaced by p.l.c., plc, P.L.C. or PLC.

Re-registration under the Companies Act does not constitute a new legal entity but merely subjects the company to certain additional company law rules.

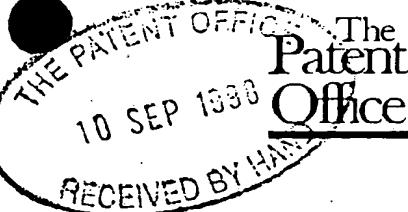
**PRIORITY  
DOCUMENT**

SUBMITTED OR TRANSMITTED IN  
COMPLIANCE WITH RULE 17.1(a) OR (b)

Signed *Andrew Gossage*

Dated 3 JUN 1999

**THIS PAGE BLANK**

11SEP98 E369427-4 D02890  
P01/7700 25.00 - 9819753.6

## Request for the grant of a patent

(See the notes on the back of this form. You can also get  
an explanatory leaflet from the Patent Office to help  
you fill in this form)

The Patent Office

Cardiff Road  
Newport  
Gwent NP9 1RH

1. Your reference

RSJ05923GB

2. Patent application number

(The Patent Office will fill in this part)

10 SEP 1998

**9819753.6**3. Full name, address and postcode of the or of  
each applicant (*underline all surnames*)The Welding Institute

Abington Hall

Abington

Cambridge

CB1 6AL

GREAT BRITAIN

774026 CO 1

Patents ADP number (*if you know it*)  
If the applicant is a corporate body, give the  
country/state of its incorporation

Great Britain

4. Title of the invention

HEAT RESISTANT PRODUCT AND METHOD

5. Name of your agent (*if you have one*)

GILL JENNINGS &amp; EVERY

"Address for service" in the United Kingdom  
to which all correspondence should be sent  
(*including the postcode*)Broadgate House  
7 Eldon Street  
London  
EC2M 7LHPatents ADP number (*if you know it*)

745002

6. If you are declaring priority from one or more  
earlier patent applications, give the country  
and the date of filing of the or of each of these  
earlier applications and (*if you know it*) the or  
each application number

Country

Priority application number  
(*if you know it*)Date of filing  
(day / month / year)7. If this application is divided or otherwise  
derived from an earlier UK application,  
give the number and the filing date of  
the earlier application

Number of earlier application

Date of filing  
(day / month / year)8. Is a statement of inventorship and of right  
to grant of a patent required in support of  
this request? (*Answer 'Yes' if:*

YES

- a) *any applicant named in part 3 is not an inventor*
- b) *there is an inventor who is not named as an applicant, or*
- c) *any named applicant is a corporate body.*

*See note (d)*

Patents Form 1/77

9. Enter the number of sheets for any of the following items you are filing with this form.  
Do not count copies of the same document

Continuation sheets of this form

Description	6
Claim(s)	2
Abstract	(1)
Drawing(s)	3 1 3

10. If you are also filing any of the following, state how many against each item.

Priority documents

Translations of priority documents

Statement of inventorship and right to grant of a patent (Patents Form 7/77)

Request for preliminary examination and search (Patents Form 9/77)

Request for substantive examination (Patents Form 10/77)

Any other documents  
(please specify)

11. For the Applicant  
Gill Jennings & Every

I/We request the grant of a patent on the basis of this application.

Signature

Date

10 September 1998

12. Name and daytime telephone number of person to contact in the United Kingdom

SKONE JAMES, Robert Edmund  
0171 377 1377

**Warning**

After an application for a patent has been filed, the Comptroller of the Patent Office will consider whether publication or communication of the invention should be prohibited or restricted under Section 22 of the Patents Act 1977. You will be informed if it is necessary to prohibit or restrict your invention in this way. Furthermore, if you live in the United Kingdom, Section 23 of the Patents Act 1977 stops you from applying for a patent abroad without first getting written permission from the Patent Office unless an application has been filed at least 6 weeks beforehand in the United Kingdom for a patent for the same invention and either no direction prohibiting publication or communication has been given, or any such direction has been revoked.

**Notes**

- a) If you need help to fill in this form or you have any questions, please contact the Patent Office on 0645 500505.
- b) Write your answers in capital letters using black ink or you may type them.
- c) If there is not enough space for all the relevant details on any part of this form, please continue on a separate sheet of paper and write "see continuation sheet" in the relevant part(s). Any continuation sheet should be attached to this form.
- d) If you have answered 'Yes' Patents Form 7/77 will need to be filed.
- e) Once you have filled in the form you must remember to sign and date it.
- f) For details of the fee and ways to pay please contact the Patent Office.

HEAT RESISTANT PRODUCT AND METHOD

The invention relates to a heat resistant product and a method for producing the product.

5 There are many applications where heat insulation and in particular fire resistance is required. Important examples include fire walls and doors for buildings and ships, ceilings, walls and internal panels of trains and other vehicles and heat shields for high performance 10 aircraft, for example leading edges of wings, noses and cones.

Various coating materials and the like have been proposed in the past to promote heat insulation in these 15 applications but these have been found to be inefficient and/or non-cost effective and there is a need to provide a much cheaper but reliable heat insulating product.

In accordance with a first aspect of the present invention, a heat resistant product comprises a ceramic binder and vermiculite, wherein between 20% and 80% of the 20 dry weight of the product is vermiculite having a particle size such that more than 80% of the vermiculite does not pass through a 2mm sieve.

In accordance with a second aspect of the present invention, a method of manufacturing a heat resistant 25 product comprises mixing mica granules with a ceramic binder; and drying the mixture, wherein between 20% and 80% of the dry weight of the product is vermiculite having a particle size such that more than 80% of the vermiculite does not pass through a 2mm sieve.

30 We have found that a very cost effective product can be obtained by utilizing a mixture of mica granules and a ceramic binder. This should be contrasted with known mica sheet made using a paper making process which is a very dense product. The new product will be much less dense 35 than this sheet material with the result that heat insulation is promoted by virtue of the trapped air. Furthermore, the resultant product is very lightweight.

making it much easier to handle and can be utilized in a variety of forms depending upon the application.

5 In one form, the product is substantially rigid and can be handled rather like wood or the like so that it can be cut to required shapes. The product is relatively brittle and so typically will be sandwiched between load supporting sheets which are adhered to the product. These load supporting sheets can comprise fibre reinforced material sheets, steel or any other material enabling the 10 panel to function as a structural or semi-structural component.

In other applications, the product is adhered to the surface of an article. Thus, a substantially rigid product could be produced which is then adhered to the article or 15 more conveniently the product is formed in-situ on the article, for example by moulding, or by spraying onto the article.

An important application for the product is with known sandwich structures. These typically comprise of a core 20 material such as foamed metals, plastics, wood, endgrain balsa or a cellular honeycomb of metal, plastic or composite materials sandwiched between planar members such as phenolic glass laminates. These sandwich structures have considerable importance within structural engineering 25 and are used extensively in aerospace and other industries since they provide key benefits over conventional materials including very low weight, high stiffness, durability and production cost savings. However, such sandwich structures do not generally operate above 170°C. If a product according to the invention is adhered to such a structure, 30 this enables the structure to be operated at far higher temperatures in view of the very low conductivity of the product.

The product can be produced in a number of different 35 thicknesses and consistency and can adhere to most surfaces including metal, wood and composites. The product can be moulded around complicated shapes and can be produced in

very thin layers which are flexible. Glass fibre, and woven glass fibre can be used to reinforce the product if required. This is dependent on the thickness of the product and the binder used.

5 In principle, any ceramic binder could be used, such a binder typically comprising an alkali silicate with a filler such as alumina, magnesia, silica or other ceramic. The preferred binder comprises the adhesive part of a two part binder such as a waterglass. Commercial examples of such a binder are Al/CS manufactured by Fortafix Limited, and Aremco 571. The Al/CS binder comprises a first part of sodium silicate and a second part of an alumina filler. We have found that a very good product is obtained by using the adhesive part only (without the filler) or possibly by 10 replacing the filler with powdered mica.

15 The step of drying the mixture could be carried out by heating or any other drying method including vacuum drying, optionally assisted by the use of microwave radiation.

20 The amount of vermiculite used will depend upon the desired density and strength of the product and the manner in which it is used. If the product is supported between sheets then up to 80% of the dry weight of the product may be vermiculite. Lower proportions would be more suitable in other applications, a preferred range being 50%-80%.

25 Examples of applications of the invention are set out below:

1. Fire walls and doors for buildings and ships.
2. Protecting surfaces that are near to a heat source i.e. exhaust manifolds on cars and trucks.
3. Cable industry where once asbestos materials were used.
4. Electronic motors, commutators, insulators.
5. Cabin and cargo holds for aircraft.
- 30 6. Ceilings, walls and internal panels of trains.
7. Heat shields on high performance aircraft i.e. leading edges of wings, noses, and cones.

8. Protection from solar or other radiant heating. Tests have shown that the heat insulating product according to the invention has a number of important properties:

- 5 1. Thermal properties, mica can withstand heat up to temperature of 1000°C.
2. Resists fire without burning.
3. Low heat conductivity.
4. Insulating electrical properties better than 10. 24kV/mm.
5. Permeability to microwaves.
6. Good resistance to arcing and arc erosion.
7. Mica is inert to most chemicals.
8. Mechanical properties are very good in 15 particular compression resistance.
9. Good tensile and bending strength.
10. Large elasticity modulus.

These properties will vary depending upon the particular mixture of mica and binder which is used and 20 these can be chosen depending upon the application.

Some examples of heat resistant products according to the invention will now be described with reference to the accompanying drawings, in which:-

25 Figure 1 is a schematic cross-section through one example of a product incorporated onto a honeycomb structure;

Figure 2 illustrates the results of a fire test on the structure shown in Figure 1; and,

30 Figure 3 is a photograph of another example of the product.

Figure 1 illustrates a honeycomb core 1 made, for example, of aluminium in a conventional manner. This core is sandwiched between a pair of phenolic skins 2,3 which are adhered to the core 1. As explained previously, this 35 sandwich structure does not have significant fire resistance and so to improve its fire resistance, a mica

heat insulating layer 4 is provided on the phenolic skin 2. This was achieved in the following manner.

5 A conventional two part ceramic adhesive Al/CS was selected and 120 grams of the adhesive part (that is a waterglass) was mixed with 2 grams of powdered mica and 26 grams of mica granules. The powdered mica took the place of the conventional aluminium filler which is normally used with the ceramic adhesive and is used in the same proportion as the filler.

10 In this example, the mica granules comprised exfoliated vermiculite with particle sizes such that 93% of the particles passed through an 8mm sieve, 32% through a 4mm sieve and 4% through a 2mm sieve.

15 The exposed surface of the phenolic skin 2 was degreased, this skin having dimensions of 150mm x 150mm x 12mm. 70 grams of an adhesive (Fortafix Al/CS)/powdered mica was applied onto the clean surface of the phenolic skin or board 2 and the board was placed in a bonding jig with the adhesive surface face up.

20 The mixture of mica granules and adhesive was applied onto the precoated surface of the phenolic skin 2 to a thickness of about 8mm. A thin layer of powdered mica was then evenly spread over the exposed surface of this previously coated mixture and the resultant product was 25 clamped over its entire area using a steel caul plate with release paper. The structure was then placed in a preheated oven for one hour at 60°C to bind the mica granules into the layer 4 and adhere that layer to the phenolic skin 2. In some cases, further improved properties can be obtained by using higher temperatures.

30 It should be understood that the provision of a thin layer of powdered mica over the surface of the layer 4 is optional as is the use of powdered mica in the earlier mixing process. It was found that the replacement of the powder filler or its omission from the binder provided a significant weight saving without adversely effecting the performance as well as providing a cost saving.

The performance of the structure shown in Figure 1 was assessed by placing thermocouples onto the structure at the positions shown in Figure 1. Series 1 thermocouple was placed on the surface of the phenolic skin 3, the series 2 thermocouple was placed between the phenolic skin 2 and the layer 4; and series 3 thermocouple was placed on the uncoated, external surface of the layer 4. As can be seen from Figure 2, the fire test showed that when a flame at 1000°C was placed on the exposed surface of the layer 4, the series 1 thermocouple indicated that the temperature on the opposite surface of the structure did not exceed 130°C for 15 minutes. The series 2 thermocouple indicates that the temperature between the layer 4 and the phenolic skin 2 did not exceed 220°C.

In view of the significant reduction in heat across the layer 4, conventional adhesives can be used to adhere that structure to the support surface.

Figure 3 illustrates another form of the product which has a substantially rigid structure and is self-supporting. This can be used as the core between load bearing boards or plates, for example made of mica (not shown). As can be seen in Figure 3, the product has substantial size and the granules are clearly visible, having a maximum dimension up to 15mm. The product can be handled in a similar way to wood as previously mentioned. This product has been formed by a moulding process and it will be clear that more complex shapes could also be achieved using appropriate moulds. This indicates the versatility of the invention.

CLAIMS

1. A heat resistant product comprising a ceramic binder and vermiculite, wherein between 20% and 80% of the dry weight of the product is vermiculite having a particle size such that more than 80% of the vermiculite does not pass through a 2mm sieve.  
5
2. A product according to claim 1, wherein 50%-80% of the dry weight of the product is vermiculite having a particle size such that more than 80% of the vermiculite does not pass through a 2mm sieve.  
10
3. A product according to claim 1 or claim 2, wherein the product is substantially rigid.
4. A product according to any of the preceding claims, wherein the binder comprises the adhesive part of a two part binder.  
15
5. A product according to any of claims 1 to 3, wherein the binder comprises the adhesive part of a two part binder, mixed with powdered mica.
6. A product according to any of the preceding claims, further comprising glass fibre reinforcement.  
20
7. A product according to any of the preceding claims, wherein the mica granules have a maximum dimension up to 15mm.
8. A product according to any of the preceding claims, wherein the product is sandwiched between load supporting sheets adhered to the product.  
25
9. A product according to any of the preceding claims adhered onto the surface of an article.
10. A product according to any of claims 1 to 7 moulded onto the surface of an article.  
30
11. A product according to any of claims 1 to 7 sprayed onto the surface of the article.
12. A product according to claim 10 or claim 11, wherein the article comprises a honeycomb structure.  
35

13. A product according to claim 12, further comprising a phenolic glass laminate sandwiched between the honeycomb structure and the product.
14. A heat resistant product substantially as hereinbefore described with reference to any of the examples shown in the accompanying drawings.
15. A fire wall comprising a heat resistant product according to any of the preceding claims.
16. A method of manufacturing a heat resistant product, the method comprising mixing mica granules with a ceramic binder; and drying the mixture, wherein between 20% and 80% of the dry weight of the product is vermiculite having a particle size such that more than 80% of the vermiculite does not pass through a 2mm sieve.
17. A method according to claim 16, wherein the drying step comprises heating the mixture, or vacuum drying the mixture.
18. A method according to claim 16 or claim 17, wherein the mixture is held in a mould during the drying step.
19. A method according to claim 16 or claim 17, wherein the mixture is coated onto a surface of an article prior to the drying step.
20. A method according to any of claims 16 to 19, wherein 50%-80% of the dry weight of the product is vermiculite having a particle size such that more than 80% of the vermiculite does not pass through a 2mm sieve.
21. A method of manufacturing a heat resistant product substantially as hereinbefore described with reference to any of the examples shown in the accompanying drawings.

# HIGH TEMPERATURE FIRE RESISTANT SANDWICH PANEL

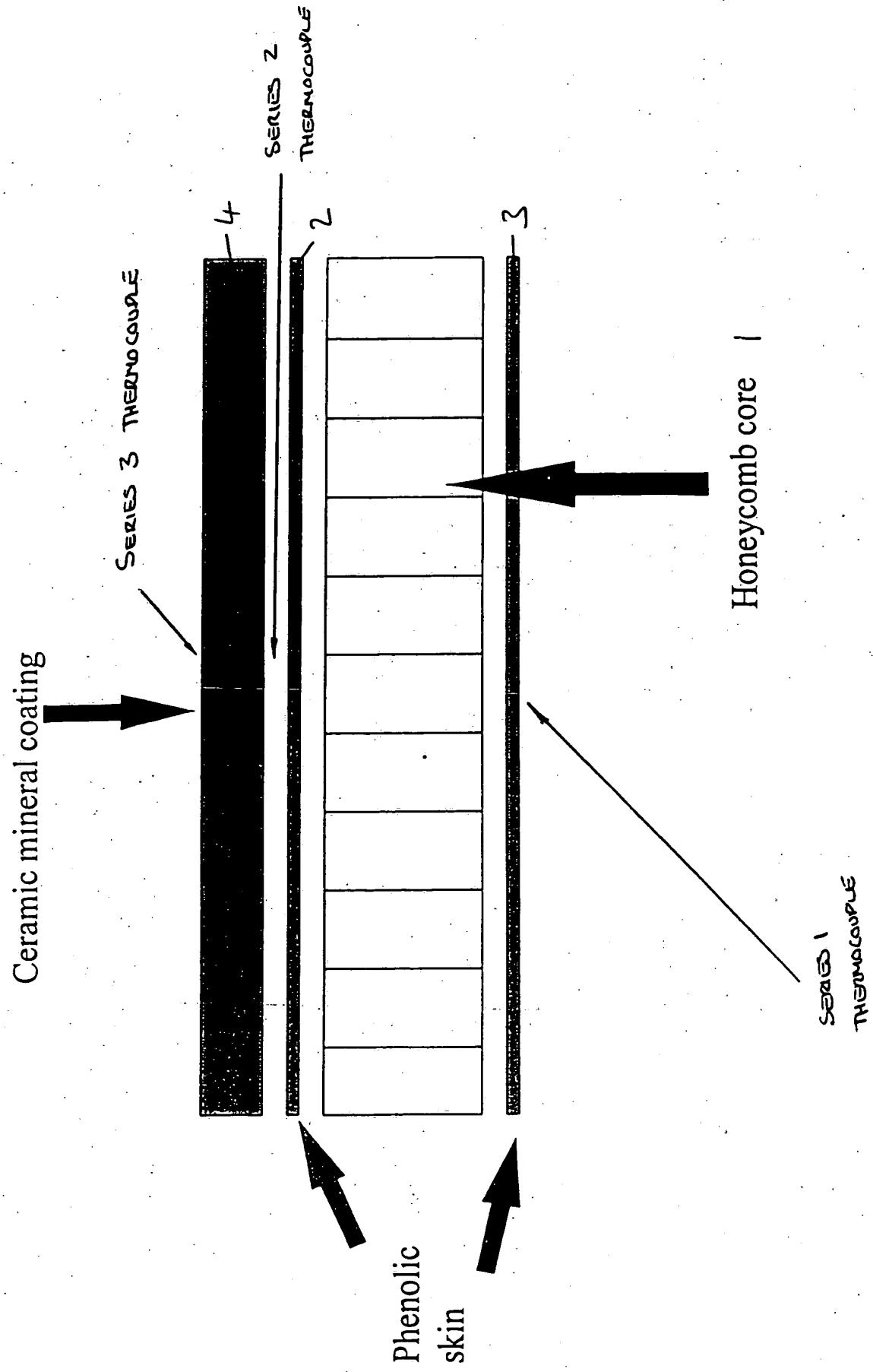


Figure 1

**THIS PAGE BLANK (USP)**

# Fire test 99692/14-1

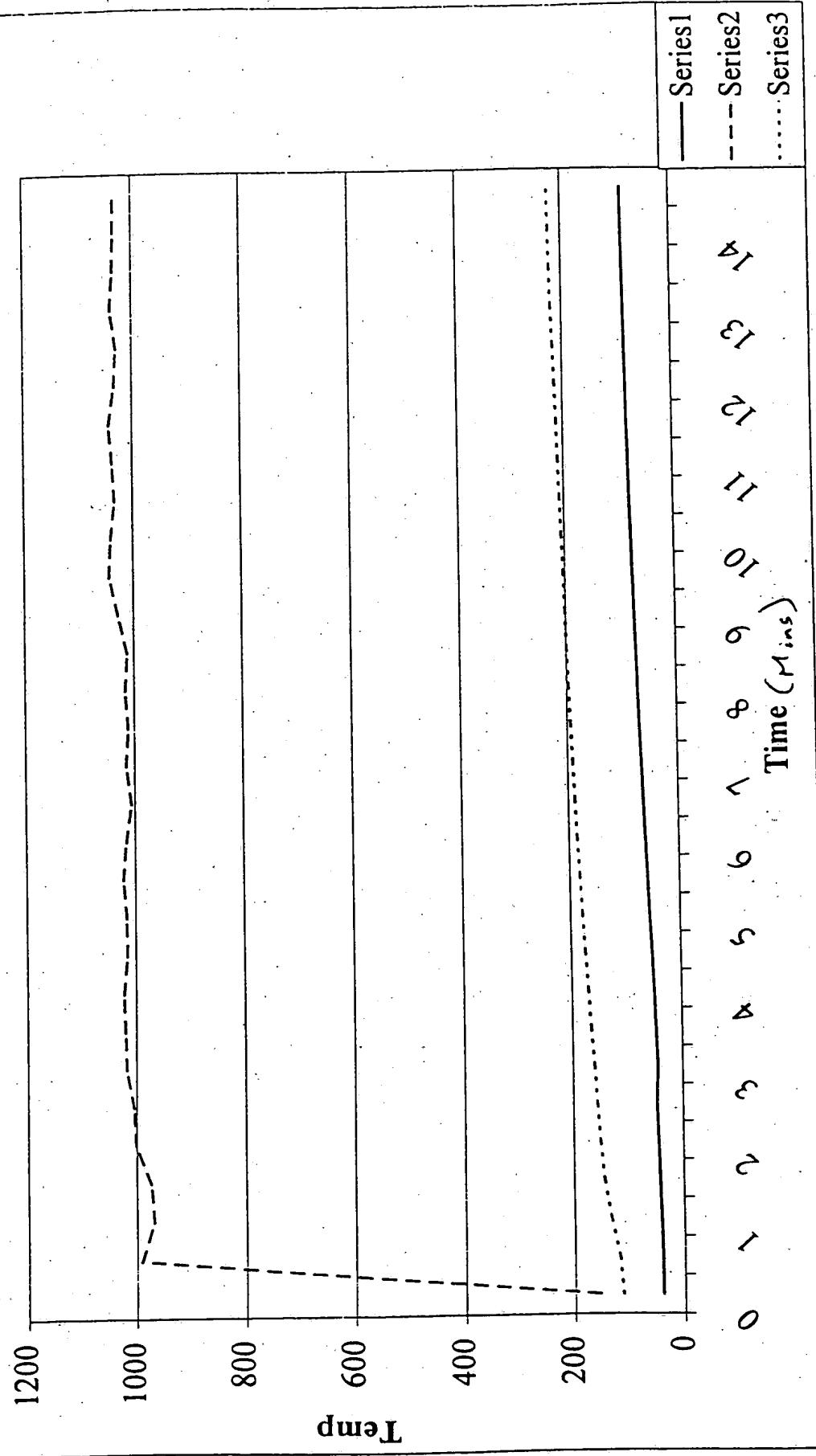
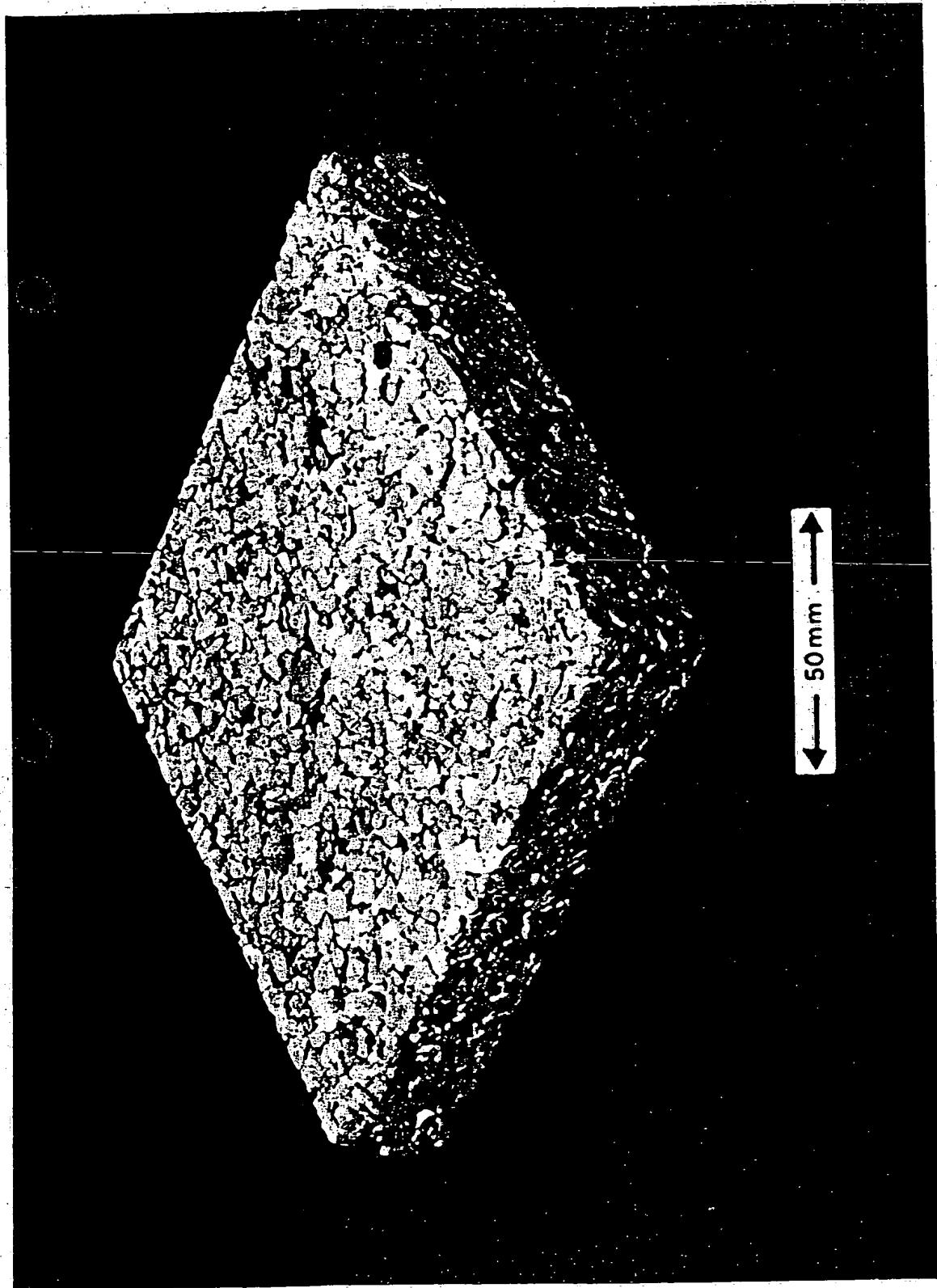


FIG 2

**THIS PAGE BLANK (USM)**



F1 6-3

PC / GCB 9901467  
Gill Jennings & Every  
1-6-99

**THIS PAGE BLANK (USP) u,**